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# Detection of $\Delta^9$ -Tetrahydrocannabinol, Methamphetamine and Amphetamine in air at low ppb level using a Field Asymmetric Ion Mobility Spectrometry microchip sensor

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## Abstract

In this work, we report the detection in air of two families of illicit drugs using a high sensitivity portable instrument based on a Field Asymmetric Ion Mobility Spectrometry microchip sensor. Methamphetamine, Amphetamine and  $\Delta^9$ -Tetrahydrocannabinol ( $\Delta^9$ -THC) were detected in the vapor phase at atmospheric pressure and a concentration of 12.5, 25 and 65 ppb, respectively. Each target molecule appears at a specific Compensation Voltage (CV) under a fixed Dispersion Field (DF). A linear FAIMS response at different concentrations and low limits of detection were successfully achieved.

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**Keywords:** FAIMS, Differential Mobility Spectrometry, THC, Cannabis, Methamphetamine, Amphetamine, Dynamic Headspace

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## 1. Introduction

Detection of illicit drugs and psychoactive substances is of increasing interest in the field of road safety due to the dramatic consequences they can have. Indeed, in cases of abuse, the driver vigilance is affected, and a large number of psychological disorders may occur [1]. Actually, the drugs consumption constitutes a real problem in the field of public security and traffic safety. It is recognized that cannabis, after alcohol, is the most commonly used drug in the

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world and it is the most frequently encountered psychoactive substance in blood of drivers involved in traffic accidents [1, 2]. As cannabis, amphetamines, which are an interesting class of psychotropic compounds, present euphoric, stimulant, hallucinogenic and other dangerous effects [2]. Usually, these kinds of compounds were analyzed by conventional techniques such as GC-MS and LC-MS/MS, but these techniques require an extensive sample preparation and the use of specific experimental conditions. Indeed, the analyses are carried on liquid samples like urine, blood and saliva [2]. In the recent years, the miniaturization of detection techniques and the realization of portable and efficient analytical systems become a challenge to reduce the analysis time and to obtain a fast and *on-site* response [3]. Recently, Differential ion Mobility Spectrometry becomes an unavoidable detection technique and knows an increasing interest in the fields of explosives and illicit drugs detection [4].

In this context, the present study was devoted to the detection of  $\Delta^9$ -THC which acts as the psychoactive substance of cannabis, Methamphetamine and Amphetamine in air using a portable Field Asymmetric Ion Mobility Spectrometry (Owlstone FAIMS technology) [5].

## 2. Principle of FAIMS microchip sensor

The detection mechanism of molecules by FAIMS includes three main steps [5, 6].

- Ionization of molecules by charge transfer: the radioactive  $^{63}\text{Ni}$  source allows first the generation of energetic electrons which interact afterward with the carrier gas (air) to form the Reactive Ion Peak (RIP) with positive and negative charges. These reactive ions are  $\text{H}^+(\text{H}_2\text{O})_3$  and  $\text{O}_2^-(\text{H}_2\text{O})_2$ . In the presence of an analyte (M) and depending on its proton and/or electron affinities, the RIP ions transfer their charge to M by collision. In this case, M replaces one or two water molecules to form monomer ions  $\text{MH}^+(\text{H}_2\text{O})_2$  and  $\text{MO}_2^-(\text{H}_2\text{O})$  or dimer ions  $\text{M}_2\text{H}^+(\text{H}_2\text{O})$ .
- Separation of ions by FAIMS microchip: using a miniaturized chip etched on silicon wafer and composed by 47 serpentine channels, the ions are separated based on their mobility under a varying Dispersion Field (DF 0 – 60  $\text{kV}\cdot\text{cm}^{-1}$ ). Ions move through channels by following different trajectories. Due to the presence of high electric field, most of the ions are neutralized. To avoid this phenomenon, a Compensation Voltage (CV) which varies between -6 and +6 V is swept across the channels. It allows ions to shift trajectories in order to reach the detector.
- Simultaneous detection of the positive and negative ions: filtered ions are simultaneously detected by a charge collector positioned in front of FAIMS microchip. Anions and cations are recorded simultaneously, which generates spectra in both polarities. Each detected ion appears at a specific Compensation Voltage under a fixed Dispersion Field (DF). The signal intensity depends on the concentration of ion in the target sample.

## 3. Materials and methods

A Lonestar 2 (LNS) instrument, based on Field Asymmetric Ion Mobility Spectrometry and three permeation ovens (OVG-4) from Owlstone, UK, were used to generate first the vapor phase and then to detect the target compounds. A mass flow controller (MFC) EL-FLOW at 5  $\text{L}\cdot\text{min}^{-1}$  from Bronkhorst – Gefran Benelux, Belgium, was used to control the diluted flow. Pure standards solutions of the target compounds  $\Delta^9$ -Tetrahydrocannabinol, Methamphetamine and Amphetamine were purchased from LGC Standards, Molsheim, France, at a concentration of 1  $\text{mg}\cdot\text{mL}^{-1}$  in methanol. Dry air was used as carrier and dilution gas. The schematic diagram of the experimental configuration was presented in Fig. 1.

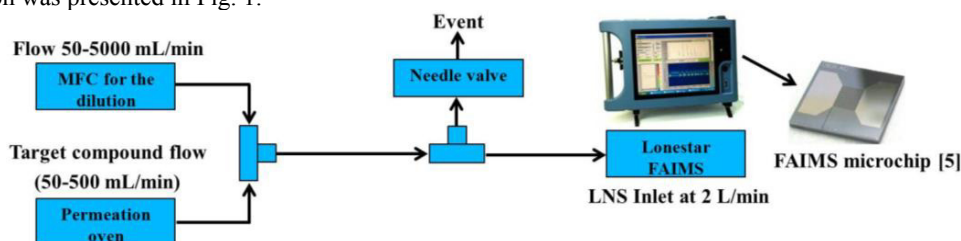


Fig. 1. Schematic diagram of the experimental configuration.

In this work, a dynamic headspace method was used to generate the vapor phase of the  $\Delta^9$ -THC. For the two amphetamines, the vapor was generated using PTFE permeation tubes.

Due to its high boiling point (390°C) and low vapor pressure ( $1.5 \times 10^{-6}$  mbar), the vapor phase of  $\Delta^9$ -THC was generated using a vial containing the standard solution (5 to 30  $\mu\text{L}$ , depending on the desired concentration) and flushed at room temperature with dry air at a flow rate of 750  $\text{mL} \cdot \text{min}^{-1}$  to the LNS inlet. In these conditions, it was possible to obtain a range of concentration between 65 to 390 ppb.

For the Methamphetamine and Amphetamine, PTFE tubes containing samples were used to generate the vapor phase by heating in the permeation oven. Permeation rates were calculated by mass loss over time at constant temperatures. After the calibration phase, Methamphetamine permeation rate at 100°C was 3385  $\text{ng} \cdot \text{min}^{-1}$ , while for the Amphetamine, it was 6005  $\text{ng} \cdot \text{min}^{-1}$  at a heating temperature of 60°C. A wide range of concentrations, between 12.5 to 250 ppb of Methamphetamine and between 25 to 500 ppb of Amphetamine, was obtained by changing the diluted gas flow rate and the sample flow rate.

The FAIMS response at atmospheric pressure for each concentration was determined as follows: the inlet flow rate was set at 2  $\text{L} \cdot \text{min}^{-1}$ , and the spectrum was obtained by changing the Dispersion Field intensity between 0 and 100% with a total analyzed time of 3 minutes.

#### 4. Results and discussion

We performed to detect the investigated psychoactive compounds in FAIMS as specific ions in the positive mode of the FAIMS spectra. The signals of these compounds correspond to the positive molecular ions and were attributed to  $\Delta^9$ -THCH<sup>+</sup>,  $\text{C}_9\text{H}_{13}\text{NH}^+$  and  $\text{C}_{10}\text{H}_{15}\text{NH}^+$  ions.

The analysis process was consisted in the determination of the blank of an empty vial by sweeping it by dry air. Then, the position of the solvent peak in the spectrum was located by sweeping 30  $\mu\text{L}$  of pure methanol by air at the same flow rate of 750  $\text{mL} \cdot \text{min}^{-1}$ . Due to the high proton affinity of methanol, it interacts with the positive reactive ions and was detected in the positive mode of FAIMS spectrum. At a Dispersion Field of 55%, the methanol signal appears at a Compensation Voltage of -2.55 V.

After these steps, the FAIMS response towards a wide range of  $\Delta^9$ -THC concentration shows that, at atmospheric pressure and a Dispersion Field (DF) of 55%, the maximal intensity of the signal was observed at a Compensation Voltage (CV) equal to 0.72 V. Using these conditions, a concentration around 65 ppb was detected in the target compound sample with an inlet flow rate of 2  $\text{L} \cdot \text{min}^{-1}$  (cf. Fig. 2-a). We noticed the presence of two separated peaks in the FAIMS spectrum which are attributed to the methanol and  $\Delta^9$ -THC molecules.

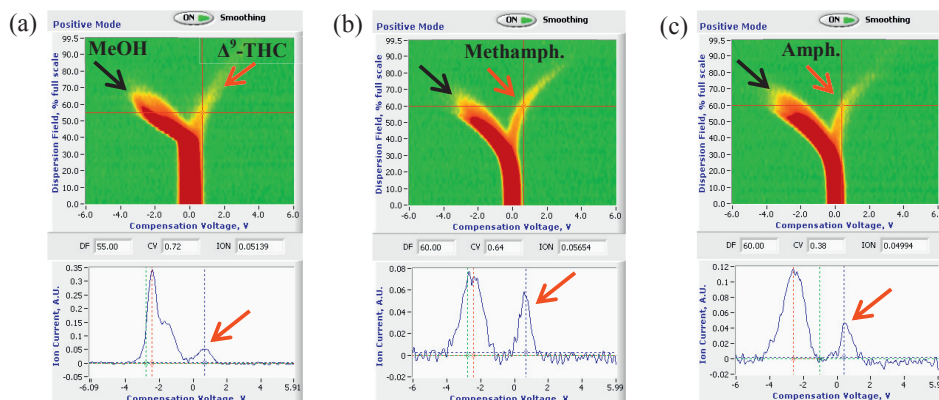


Fig. 2. FAIMS spectrum of (a)  $\Delta^9$ -THC at 65 ppb; (b) Methamphetamine at 12.5 ppb; (c) Amphetamine at 25 ppb.

For the amphetamines, we followed the same experimental procedure. So, the blank spectrum of an empty permeation oven cell was determined. After calibration of a methanol permeation tube at 60°C, its proper FAIMS spectrum was obtained at a concentration of 6 ppm. This allows identifying the position of the methanol signal using these experimental conditions, by comparison with the dynamic headspace method.

For the Methamphetamine and Amphetamine, the determination of their specific signals was carried out by detecting each component separately at increasing concentrations.

The results show that, at the same DF of 60%, the maximal peak intensity of Methamphetamine and Amphetamine appears at CV equal to 0.64 and 0.38 V, respectively. In terms of sensitivity, the limits of detection for the Methamphetamine and Amphetamine have been estimated to 12.5 and 25 ppb, respectively (cf. Fig. 2-b & c).

As in the case of  $\Delta^9$ -THC, the FAIMS sensor shows a good selectivity between the methanol and the amphetamines peaks. In the positive mode of FAIMS spectra, a fingerprint signal was located at a specific CV for each target compound.

For both psychotropic families, low limits of detection and a good repeatability of analysis were obtained by the FAIMS technique. We also demonstrated that the FAIMS response towards  $\Delta^9$ -THC, Methamphetamine and Amphetamine at different concentrations is linear as shown in Fig. 3.

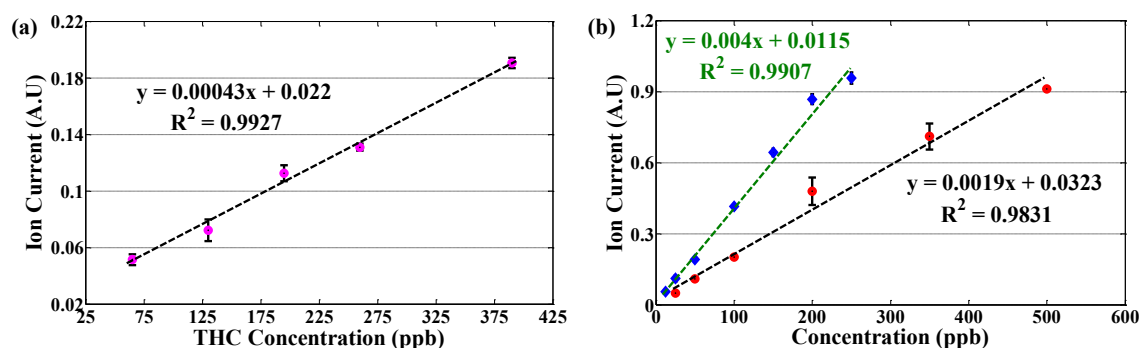


Fig. 3. Limits of detection of FAIMS sensor towards (a)  $\Delta^9$ -THC; (b) Methamphetamine (blue points) and Amphetamine (red points).

We noticed that the peak intensities increase with the concentrations which reflect the detection of an increasing numbers of the positive ionized molecules. Also, we showed that even at high concentrations, the saturation phenomenon of the detector has not been observed, so that reliable of detection capabilities can be expected in complex samples.

## 5. Conclusion

FAIMS microchip sensor allows a fast and sensitive detection of two types of psychoactive compounds in the air at low ppb levels. In presence of methanol as solvent, two peaks perfectly separated were obtained in FAIMS spectrum. Indeed, each target compound appears at a specific Compensation Voltage under a fixed Dispersion Field. The results demonstrated the potential of FAIMS instrument to be deployed as a portable and fast detection device in the field of public security. Finally, the next step of this work will be the implementation of a chromatographic micro-column to improve selectivity in order to control the exalted breath of abuses.

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